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## APPLICATION OF QUANTITATIVE MICROSCOPY TO CERAMICS

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Yasuhiko Kondo

## 1. Introduction

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The method of microscopic analysis of substances is being used in fields such as metallics, minerals, etc., and it is anticipated that it will be applied to more studies in the future. However, only the surface of the subject to be analyzed can be microscopically observed and there is only a chance that this surface will give a cross section of the substance. There has consequently been some resistance to discussing the properties of the substances based on this structure. However, there have been many studies of substances based on 1 micrograph and very accurate conclusions have been reached.

It appears that surface observation using microscopes will be reduced when it becomes possible to observe the inner structure of opaque substances by another technique. However, optical microscopes are still very important, even though X-ray permeation methods and scanning electron microscopes have been developed. Therefore, the development of a technique that can estimate the 3-dimensional structure of a substance, based on the two-dimensional information obtained from the material surface for cases where it is impossible to observe the inside of the material, is being considered. That is, the subjects that we observed gave 2-dimensional images where the 3-dimensional inner boundaries of the materials intersected the cross-sectional plane completely by chance. The form and size of the images were only part of the actual 3-dimensional image. Therefore, a science for estimating the 3-dimensional space based on the two-dimensional structure, which can be observed, was developed. This is generally known as stereology. The quantitative study of structures

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\* Numbers in margin indicate foreign pagination.



(quantitative microscopy) was developed as a result. Then Elias et al., established the International Association on Stereology in 1961.

On the other hand, the method whereby changes in the structure during image conversion are traced was proposed as a result of mathematical morphology studies on two-dimensional surfaces as a method for quantitative study of structure [2]. This method is introduced in the report of Takaku et al. [3].

In this report we describe the analysis of graphite structures in cast iron by a case study procedure while focusing on the application of quantitative microscopy to ceramics.

## 2. Cast Iron

It will be necessary to briefly explain what cast iron is before proceeding with this report. Cast iron is an iron alloy that is generally called "cast metal." It can be used in various fields, such as industrial equipment, automobiles, etc. This usually refers to an iron alloy that contains approximately 2% or more of carbon in an iron-carbon two-element alloy. However, the carbon content of cast metal that is generally used is 2.5-4.5%. In addition to iron and carbon, the metal may also contain silicon, manganese, phosphorus, sulfur, etc. Cast iron has good fluidity in liquids and the rate of contraction with hardening is low. Therefore, it is a metal that is suitable for casting complex forms. Japan produces approximately 4,000,000 tons per year.

Graphite or a carbide ( $\text{Fe}_3\text{C}$ ) is crystallized by solidification of the carbon in the cast iron. However, this explanation will be restricted to the case of graphite crystallization. Graphite that has been crystallized by cocrystallization and solidification has an effect on the chemical structure and the solidification conditions. Its shape, size and distribution vary and affect the mechanical properties, thermal properties, and other

properties of the cast iron. For instance, the tensile strength of cast iron changes from  $10 \text{ kg/mm}^2$  to  $60 \text{ kg/mm}^2$  with changes in the form of the graphite. Consequently, quantitative determination of the graphite structure is an important problem from the viewpoint of research on cast iron. However, many of the attempts to study the form, size and distribution of the graphite have been restricted to explanations with standard figures.

Figure 1 and Figure 2 show the classifications of graphite pertaining to the form and distribution of the substance respectively [4]. Those pertaining to size are classified in 8 stages. The actual structure of graphite in cast iron is represented with these classification figures as the comparative standards. The tendency to represent these as quantitative figures has increased suddenly with the appearance of image analyzers.

The details that will be introduced here are also the result of research carried out under this condition. It must therefore first be understood that it will take more time to completely quantitatively analyze graphite structures.

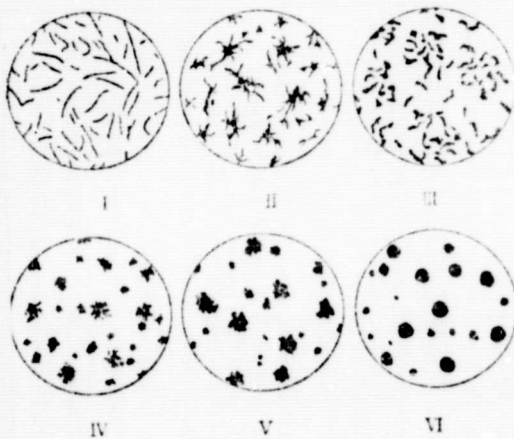


Figure 1. Form of graphite

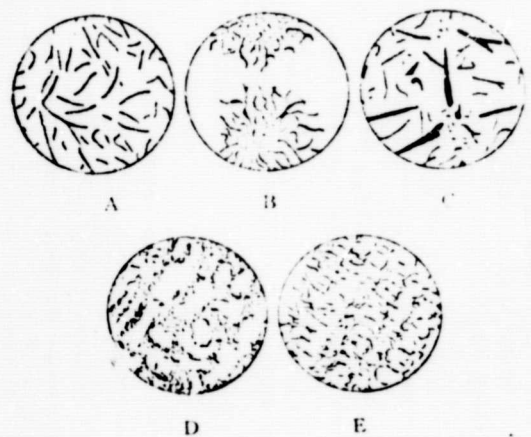


Figure 2. Distribution of graphite.

Since the properties of cast iron are determined by the graphite content of this cast iron, it is necessary to analyze the graphite structure. However, it appears that the pores in the sintering process and original powder should be explained to the readers.

The methods for analyzing the structure of graphite that have been reported thus far can be classified as follows.

- (1) The method whereby the properties of the structure are determined by combining each parameter [5-7]
- (2) The method whereby stereoscopy is employed [8-11]
- (3) The method whereby image conversion is employed [10, 11, 12-13]
- (4) The method whereby similar patterns are used [16,17]
- (5) Other methods [18-20]

Of these, the methods in (1) - (3) will be treated in this report. However, we will also report on the other methods using image analyzers. Image analyzers consist of TV cameras that are observed by humans. Thought processes are carried out by computers. There have been many recent developments in electronic technology and many types of devices are being marketed. The theory for measurement using the image analyzers is the spot analysis method. The micrograph structure equipped with a TV camera is introduced as an image and a monitor. The outline corresponding to the concentration (called gray value) of the structure appears on a monitor using the intersecting points (called image points) of the TV scanning rays. The object to be measured is thereby made into an image. Moreover, the structural parameters that can be measured are classified into the four types of surface area, size, peripheral length, and number

of particles. It is possible to determine various sizes for directional particles. Figure 3 shows one example of parameters measured that pertain to the size of directional particles. However, there are various parameters for size and it is obvious that the parameter must be selected in accordance with the purpose of the study.

Furthermore, the structure to be studied is selected by the gray value and measured after it appears on the TV monitor as an image. However, prior to this, tests should be carried out to determine the best image for the parameter to be measured. For instance, during analysis of the particles or pores, the measurement masks should be removed. Moreover, when foreign matter is observed in the pores, this should be removed. In addition, when particles that do not meet the purpose of the tests according to their gray value are selected, they should also be removed. These processes must be carried out in order to remove the unnecessary images (called image treatment). These processes are determined by evaluations by specialists on the measured materials. However, the image treatment method for each material is a problem that must be settled by each field.

In addition, there is a method whereby individual measurements of each particle can be carried out while simultaneously measuring the particles as a whole. The former is effective for cases where the form, surface area, and size distribution must be determined and the latter is effective for surface area ratio measurements and stereoscopic measurements.

The results were studied by the above-mentioned analytical method taking the use of an image analyzer into consideration.



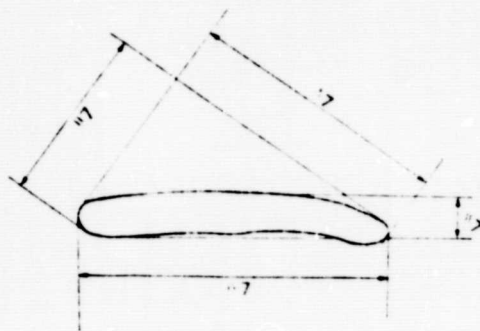


Figure 3. Size of Directional Particles.

### 3.1 Method for Determining the Features of the Structure by Combining the Parameters

This method is used for analysis of graphite forms. The technique is similar to analysis of forms of powders used in powder engineering. Removal of graphite microparticles, removal of particles on the measurement mask and removal of impurities inside the graphite are carried out. The surface area (A), size (L), and peripheral length (P) of each particle are determined. These parameters are then combined and expressed as the form of the image in image treatment.

Table 1 shows the nondimensional values pertaining to graphite forms currently being proposed. Of these, the form coefficient becomes smaller as the graphite form breaks down with a true circle being 1. These values were determined with the circle being the standard form.

The spherical ratio is the graphite surface area per surface area of the circle surrounding the graphite. Those with a spherical ratio of 80% or more are called spherical graphite according to the method for evaluation of the spherical ratio by the Japan Society on Minerals [21]. The above-mentioned two amounts should be applied to graphite that is theoretically or substantially spherical. However, when applied to the first graphite shown in I-III of Figure 1, there is a chance that the physical significance of the values will be unclear.

TABLE 1. NONDIMENSIONAL VALUES FOR GRAPHITE FORM EVALUATION BY COMBINING STRUCTURAL PARAMETERS

Nondimensional value	Computation equation	Measurement
Form coefficient	$K=4\pi (A/P^2)$	Surface area, peripheral length
Spherical ratio	$S=A/(\pi L_M^2/4)$	Surface area, maximum length
Axis ratio	$R=L_W/L_M$	Width, maximum length
Eccentricity	$E = P/2) / \sqrt{L_H^2 + L_V^2}$	peripheral length, projection length
CV ratio	C: ratio of spherical particles to flat particles	particle number

The axial ratio is the ratio of the length of the graphite to its width. Spherical graphite has a value of 1. This value becomes smaller as the graphite becomes more slender. This value can be determined relatively easily from the micrographs without an image analyzer.

Eccentricity is a value that was proposed as a measure of the degree of branching of the graphite [5]. Unbranched flat graphite has a value of 1. This value increases as the number of branches increases.

The CV ratio was suggested as a measure for evaluating the form of graphite having a shape somewhere between spherical and flat (corresponds to III in Figure 1, called CV graphite; cast metal that has been focused on in recent years) [6]. The form of graphite with an intermediate shape is evaluated from the ratio of the number of graphite particles with a spherical ratio of 60% or more and the number with a spherical ratio of less than 60%.

Figure 4 is an example of form analysis of cast metal having the various graphite structures in the figure using the form coefficient (K), the spherical ratio (S), the axial ratio (R) and the CV ratio (C) in Table 1. The four graphite structures to the left of the figure are CV graphite and the two to the right are spherical graphite. The graphite structure becomes more massive in form as we move to the right of the picture. There is a tendency for each measurement value to correspond with the changes in form. Of these values, K shows relatively continuous changes in comparison to the other nondimensional forms and it can be said that this is an efficient value for classification of graphite forms that vary as flat CV and spherical graphite. However, there is a problem with the use of this measure with flat forms. Moreover, each value in Figure 4 is the mean value for individual graphites. However, there are cases where the graphite forms change within the field of vision (and within one cross section). The standard deviation of the mean values is therefore large. When these forms are represented only by the mean value, there are often various shapes represented by the same value. Therefore, tests were carried out on the representation of graphite forms with not one mean value, but two values. An example is shown in Figure 5 [5]. Figure 5 classifies the graphite forms by their form coefficients and axial ratios. It can be said that this is more effective than the use of mean values. /136

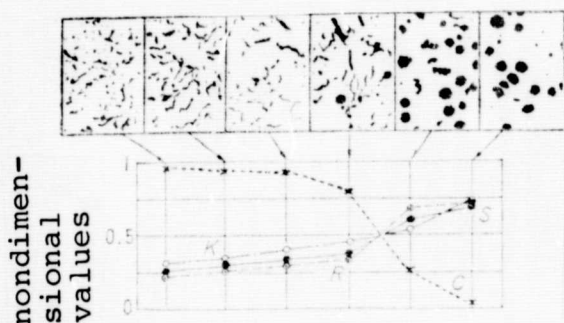


Figure 4. Form Analysis of Graphite by Nondimensional Values (Table 1)

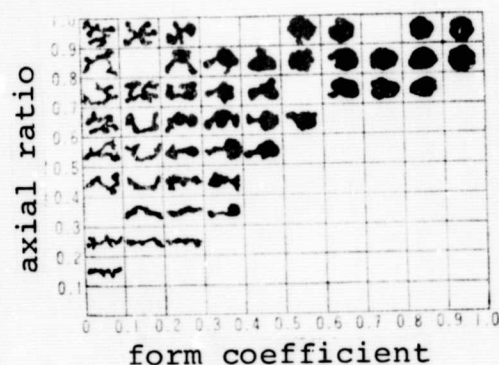


Figure 5. Evaluation of Graphite Forms by Two Nondimensional Values

However, a simpler method is preferred for structural treatment of industrial materials such as cast iron. Although a complex method is obviously more precise, it is difficult to apply. This problem is the same as that found in ceramics and will be studied in the future. However, the purpose of structural qualitative measurements is not to numerically determine the structure alone. The ultimate purpose pertains to how these values are related to material properties, or that is, control of material properties by the structural values. It appears that it will become easier to develop a simple structural analysis method as research proceeds taking this point into consideration.

### 3.2 Method that uses stereoscopy

The measurements in the previous section were carried out in order to represent the form of two-dimensional images that appear from cast iron as basic forms. This will give the form of the graphite a nondimensional value, as can be done in concrete terms for the diameter of circles and spheres. However, there is no guarantee that the values will be uniform in solid spaces of material whose surface is being observed. For instance, even though the form can be accurately represented, there are still problems with the method. However, we can say that this method is suitable for the case where simple particles are used and the form is to be determined, as in the case of powders.

On the other hand, stereoscopy is the method whereby the values of graphite present in solid spaces are estimated from information obtained from points and lines of the lower dimension. It is one technique for qualitatively treating the form of materials [1]. This technique is a geometric probability theory and dates back to references from 1848.

Stereoscopy is applicable to the case of particles and crystals of various sizes with similar shapes that are arranged at random. Special treatment is necessary for the case of



particles that are oriented while being maldistributed. Osawa has introduced the explanation of Elias as follows as a subject of stereoscopy [22].

(1) Form and Number

When many particles or cells of a similar shape are present in the material, the number of particles present per unit volume and the average form are estimated.

(2) Capacity

The volume percentage occupied by a certain phase is determined.

(3) Size

The particle size and distribution are determined.

(4) Surface Area (Interface Area)

The interface area per unit volume of the material is determined.

(5) Length

The total length of the fiber structure, crystal structure, etc. in the material per unit volume is determined.

(6) Spatial Distribution of Particles

The contact percentage between particles, the number of particles that make contact, the contact surface area, and the distance between interfaces is estimated.

(7) Anisotropism

Orientation of the particles and cells is estimated.

(8) Analysis when the Sample Does Not Have a Random Structure

Analysis is carried out when the sample has an oriented structure or when several aggregates of particles are formed.

(10) Continuous Cross Section Micrograph Technology

A sample is cut along parallel cross sections at uniform distances and each cross section structure is analyzed.

## (11) Research and Stereoscopy Equipment

Research should be carried out on equipment and gauges and computers should be automated for rapid and accurate retrieval of quantitative information (for instance, number, length, surface area, etc.) from images of the cross sections.

Figure 6 summarizes the correlation between the basic parameters that have been established thus far and the three dimensional values [23]. In the figures, P is a point, L is the line, A is the surface area, S is the surface area, and V is the volume. The large letters show the amounts pertaining to graphite and the added figures show the amounts pertaining to measurements. For instance,  $P_p$  shows the value obtained by subtracting the number of test points inside the particle from the total number of test points during the measurements. According to the basic equation listed in the figure,  $P_p$  is equal to the surface area percentage ( $A_A$ ) or the volume percentage. There have been many references that have listed this basic equation previously [1,23, 24-27].

Since the stereoscopic method is based on the geometric probability theory, it seems that the sample to be measured should have a structure common to samples in general and that it is necessary to determine the measurement field for a standard deviation because there is always error in probability theories. The image analyzer was therefore used. Table 2 shows the definitions of the symbols used in Table 2.

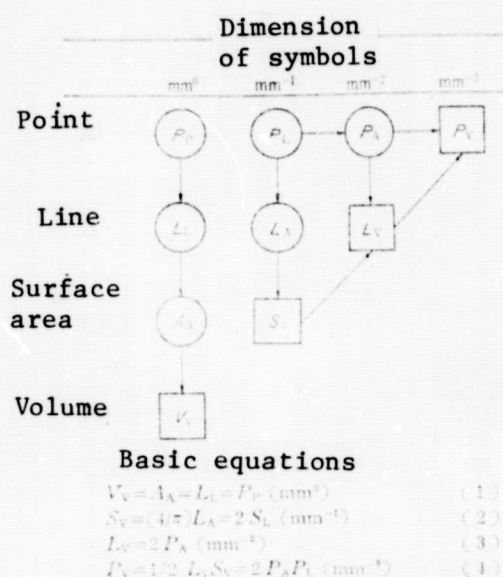


Figure 6. Correlation between Measured Values (O) and (□) in Stereoscopy and Basic Equations

TABLE 2. DEFINITION OF MAIN STEREOSCOPIC SYMBOLS

Symbols	Definitions
( $P_p$	Value obtained by subtracting the total of test points that fall into the particle to be measured from the total test points at the measurement site (point percentage).
( $P_L$	Value obtained by subtracting the total of intersections between particles and test lines from the total length of the test lines.
( $P_A$	Value obtained by subtracting the number of points where the line elements intersect the cross section from the experimental cross section area.
( $L_L$	Value obtained by subtracting the total length of test lines cut by particles from the total length of the test line (line percentage)
( $L_A$	Peripheral length of particles per unit cross section area
( $A_A$	Area of particles per unit cross section area (percentage).
( $L_V$	Length of line elements in unit volume
( $S_V$	Surface area in unit volume (specific surface area)
( $V_V$	Volume of particle in unit volume (volume percentage).

Therefore, we will explain the use of stereoscopy for analysis of graphite structures in cast iron using the model shown in Figure 7. Figure 7 is an example of the structure of cast iron with spherical graphite. This is the case where a 5 mm square lattice was used. The square lattice can be made by any method. Moreover, the total length of the scanning lines and the points of intersection of the scanning lines are already known. When the points of intersection that fall to inside the graphite particles are determined,  $P_p$  is the volume percentage or area percentage and when the total length of the scanning lines that cut inside the graphite particles are known,  $L_L$  can be determined. Therefore, the area percentage or volume percentage are known. On the other hand, there are 68 points where the scanning lines are crossed. Therefore, the cutting point  $P_L$  per total length of the scanning lines is  $68/14.4=4.72$ . The dimension at this time is  $\text{mm}^{-1}$ . When  $P_L$  is known, the specific surface area  $S_V$  of the graphite in the solid can be determined from the basic equation in Figure 6. That is, since  $S_V=2P_L$ ,  $S_V$  is  $9.44 \text{ mm}^{-1}$ .

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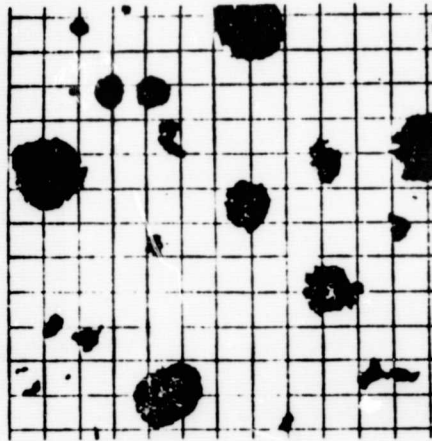
When  $S_V$  is known, the specific surface area  $S_{VP}$  per 1 particle, the mean free distance (between particle ends)  $\lambda$  of 3-dimensional particles, and the spherical diameter  $D_{3S}$  of 3-dimensional systems can be determined.

$$S_{VP}=S_V/V_V=S_V/A_A \quad (5)$$

$$\lambda=4(1/S_V-1/S_{VP}) \quad (6)$$

$$D_{3S}=6/S_{VP} \quad (7)$$



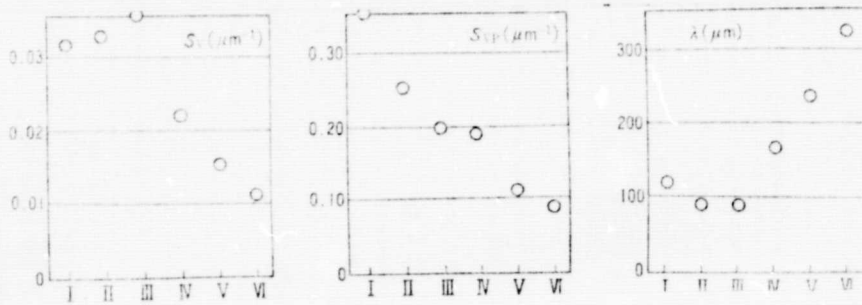


Total length of scanning line  $L_T = 14.4 \text{ mm}$   
 Total points of intersection of scanning line  $P_T = 169$   
 Points of intersection that fall into graphite  $P_1 = 20$   
 Points of graphite cut by scanning lines  $P_2 = 68$   
 $P_T/P_1 = 0.118$   
 $P_2/P_1 L_T = 4.72 \text{ mm}^{-1}$

Figure 7. Model for Analysis of Spherical Graphite by Square Lattice

Figure 8 shows  $S_V$ ,  $S_{VP}$  and  $\lambda$  determined from the above-mentioned method for the forms of graphite in Figure 1. According to the figure, of the classifications, the  $S_V$  and  $\lambda$  of Graphite I-III, which are flat graphites, can be clearly separated from the massive IV-VI graphites. Moreover, the  $S_{VP}$  changes continuously with changes in the graphite form. Therefore, it appears that these parameters can be treated as common parameters of form.

The relative surface area of the graphite is effective for classifications of graphite forms. However, the specific surface area does not show the concrete form of particles in solid. There are no values that can clearly determine whether the graphite in the case iron is spherical or flat. They are parameters that are common to graphites of each type of form. When we consider the fact that new information can be obtained on the material properties based on parameters common to the different structures, it appears that qualitative analysis of the structure of the materials is not effective for concrete determination of form.



I-VI: classification symbols for graphite forms

Figure 8. Analysis of graphite form classifications.

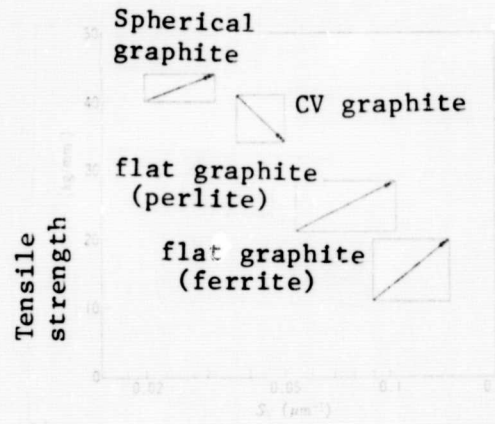


Figure 9. Correlation between specific surface area  $S_v$  and tensile strength of graphite.

Figure 9 shows an example of the statistical determination of the specific surface area  $S_V$  of graphite in cast iron by the image analysis method and the correlation with the tensile strength of the cast iron. According to the figure, the strength of cast iron decreases with changes in form from spherical, CV, and flat graphite and the strength of the cast iron increases with spherical and flat forms with graphite of the same form. However, it decreases in graphites of the same form with CV graphite. The reduction in strength of CV graphite depends on changes in form and the increase in strength with spherical and flat graphite depends on the fineness of the graphite. That is, it is clear that the specific surface area and size are a major factor in strength. This is therefore very effective in graphite structural analysis of cast iron.

Moreover, research is also being carried out on analysis of particle crystallization, particle surface area of pure alloys, changes in products formed from the particle boundary, the interface area of cocrystallized alloys, the  $S_V$ , form and size of the graphite, etc. in many fields of metals.

### 3.3 Method Using Image Conversion

The explanations given thus far have been measurements of samples whose images have been obtained from micrographs. However, the method has been recently developed whereby new information can be obtained by converting the image obtained and tracing its structure parameters during this process. Serra [2] proposed mathematical morphology as a new morphology and a method has been developed whereby the measurement site and image are converted using the correlation between various theories based on the fact that the image and measurement site are made from an aggregate of points. The results have been used in the development of the Texture Analysis System of the Raitsu Co. The authors have also used this method.

The theory of image conversion has been previously explained [2] and will therefore be omitted from this text. However, the following analysis results obtained from image conversion will be explained.

There are basically four types of image conversion.

(1) Erosion  $X \ominus B = \{x: B \subset X\}$

Here X is the image, and B is the structural element.

All are dot aggregates. X shows the individual points.

(2) Expansion  $X \oplus B = \{x: B \cap X \neq \emptyset\}$

Here 0 is a space aggregate

(3) Opening  $(X \ominus B) \oplus B$   
 $= \{x: B \cap (X \ominus B) \neq \emptyset, x \in B, X\}$

Here B is the aggregation of spectra opposite B.

(4) Closed  $(X \oplus B) \ominus B$   
 $= \{x: B \cap (X \oplus B) \neq \emptyset, x \in B, X\}$

The structural elements are used for conversion of the image. The image is either lost (erosion) or becomes larger (expansion). Hexagonal forms, line components or pairs of points made into aggregates should be used according to the measurements.

Figure 10 explains the theory of erosion conversion. When original image X at the top is eroded by structural element B (a circle is used for convenience; however it is actually a hexagonal form), large particles remain, as can be seen in the lower left of the figure, and two small particles are removed. On the other hand, when a pair of points is the structural element, only particles corresponding to the distance between the pair of points remain and the others are removed (lower right of figure). Therefore, although the images converted by the form of the element differ,



in the former case the information that is obtained pertains to size and in the latter case the image is on distribution. Figure 11 shows the case where the image that was eroded by the round structural element in Figure 10 expands with the structural element for size and then returns to its original shape. Image conversion is open conversion whereby after erosion, expansion is immediately carried out.

It is obvious that a new analysis can be carried out using the image conversion operation. In this case it is necessary to clearly define the structural elements. An image analyzer is being marketed which combines the process of image conversion with new electronic technologies. We would like to focus on this analyzer. The structural elements are point aggregates and these aggregates correspond to the original point of a mathematical coordinate. They must be symmetric to the original point and their size can be changed.

It is assumed that erosion conversion of certain phases or of pores during the sintering process occurs. The erosion process can be explained with the square model shown in Figure 12 with the pair of dots and line components being the structural elements. The figure is a model where the square particles that are 8 mm on one side are arranged so that they are approximately 2.3 mm apart with 4 horizontal lines and 3 vertical lines. The pairs of dots are shown in the top of the figure. The bottom shows conversion when the images have been eroded by the line components (original point to left end in each case). The distance between the pair of points ( $h$ ) and that of the line components ( $l$ ) increases by distance between image points  $da=0.222m$  mm. The amount of erosion increases. An eroded image of  $1-10da$  is eroded uniformly from the right with 12 individual images (2.22 mm). However, the image eroded by a pair of points is also the same with regard to length. When  $l$  and  $h$  increase with the distance between particle, for instance when  $l=h=20da$  as in the figure, a new image appears at the top and there is a difference between

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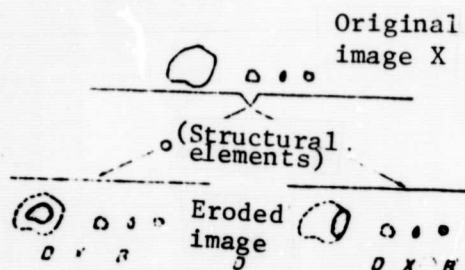


Figure 10. Erosion conversion model in case where Circle (B) and pair of points (B') are used as structural elements.



Figure 11. Open model by expansion conversion of eroded image in Figure 10.

images eroded by different structural elements. The length exceeds the size of the particles when  $h=1=37da$ . However, in contrast to the fact that 9 particles remain at the top, the particles have been removed from the bottom. The changes in area of each image during changes in the images in Figure 12 were measured and the correlation between the structural elements and the area percentage is shown in Figure 13. The area percentage of the image eroded by a pair of points is shown in the figure as  $C(h)$  (called covariance because the form of the function is the same as the statistical covariance function) and by  $P(1)$  in the case of linear components. According

to the figure,  $C(0)=P(0)$ . This is the area percentage of the square particle image in the measurement region.  $C(h) \geq P(l)$  when  $l=h \geq 6 da$  ( $da=0.222$  in Figure 12; however,  $da=0.444$  in Figure 13).  $P(l)$  decreases linearly and  $l=18da$ . That is  $(P_{91}) = 0$  with a length of 7.99 mm. Therefore, the projected image length of the square particle is given. When  $l$  and  $h$  are small,  $P(l)=C(h)$  and  $P(l)$  decreases linearly. Therefore, these functions are uniform with  $l=h=0$ . Consequently,  $P(l)$  can be substituted with measurements of  $C(h)$ .  $P(l)=0$  when  $l > 18 da$ . On the other hand, covariance  $C(h)$  becomes smaller with a reduction to  $h \geq 6 da$ . Nevertheless, this shows the distance between the ends of each particle. The smallest points are shown by  $h=17 da$ . There is an increase up to  $h=23da$ , where the largest points are shown. Then the same changes are repeated and  $C(h)$  shows the periodic properties. The distance of 10.21 mm from 0 to the maximum  $h$  is 23 da shows the distance between the center of each particle.

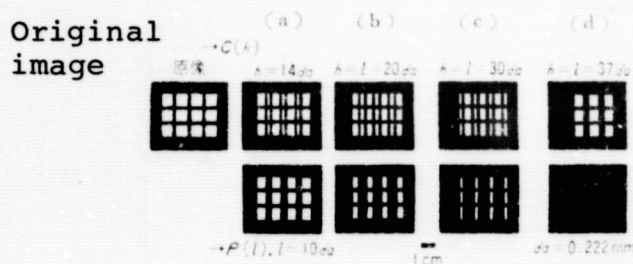
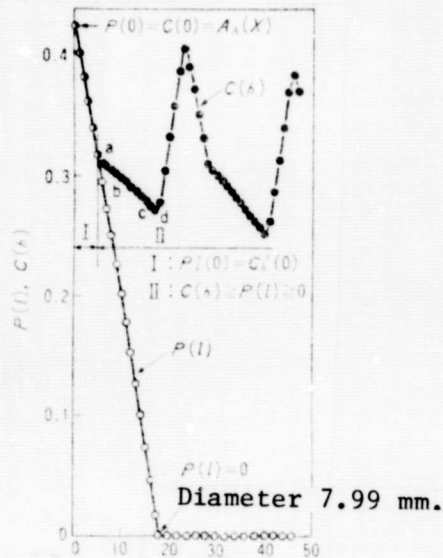


Figure 12. Eroded image of square model (8 x 8 mm, 4 lines x 3 ross) (top: pairs of points, bottom: line components)

From the above-mentioned it is clear that the information obtained from continuous line components or from pairs of points distributed spatially differs even with the same one-dimensional structural element. Information on the size of the particles is obtained from line segment structural elements and information on the distribution of particles is obtained from structural



Dimensions of structural elements (x 0.444 mm)

The a,b,c and d in the figure correspond to the (a),(b),(c), and (d) in Figure 12.

Figure 13.  $P(l)$  and  $C(h)$  of Square Models

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elements consisting of pairs of points. Moreover, when these measurements are taken in various directions, information is also obtained on the anisotropism of the particles and anisotropism of the distribution.

Covariance analysis can be used for distribution of flat graphite, anisotropism, analysis of the form of the graphite, measurements of the spacing of dendrite structures, analysis of the layer structure and distribution of the clusters.

Next, we will describe image conversion by hexagonal forms as two-dimensional structures. There are often cases where erosion or expansion by hexagonal figures is used as a means of image conversion mainly for removal or aggregation of the image.



Figure 14 is the case where open and closed image conversion has been carried out by hexagonal structural elements using spherical graphite. During the open phase the original image is first eroded by the structural elements, as was previously mentioned. When small particles are removed by erosion, the large particles disappear from the outer edge. Next, the image is expanded and the particles that disappeared revert to approximately the same image as was initially used. When this process is repeated while gradually increasing the size of the structural element, the particles are gradually removed in accordance with the size of the structural element, as can be seen in Figure 14(s). All of the particles are eventually removed. On the other hand, during the closed phase the original image is first expanded and then eroded. Image conversion is carried out by repeating this process while increasing the size of the structural element. The adjacent particles are gradually connected to form an aggregate.

The open phase is similar to the process whereby particles are treated by sifting. Information is obtained on the size of the particles. The closed phase is used to obtain information about the correlation between adjacent particles, that is, distribution of the particles.

Analysis of size distribution by the open method will be explained as an example of obtaining information on form. When changes in the area percentage or number of particles are measured during the image conversion process in Figure 14, the area percentage in Figure 15 is obtained. In the figure the area percentages for the cases of erosion, expansion, and a closed phase are also listed. The area percentage by each conversion is as follows. /142

$$A_{\text{erosion}} < A_{\text{open}} < A_{\text{original image}} < A_{\text{closed}} < A_{\text{expansion}}$$

When changes in the area percentage of image conversion by the open phase are shown by  $A_A[X_H(r)]$  and this is represented by  $\Omega_H(X;r)$ ,

$$\Omega_H(X;r) = A_A[X_H(r)] \quad (8)$$

Thereupon,  $H(r)$  indicates the structural element and  $r$  represents the size of the element.  $X_{H(r)}$  therefore shows the image converted by  $H(r)$ .

Based on the above-mentioned equation, the function pertaining to the particle distribution is defined as follows.

$$G_{H(X; r)} = \frac{\Omega_H(X; r=0) - \Omega_H(X; r)}{\Omega_H(X; r=0)} \quad (9)$$

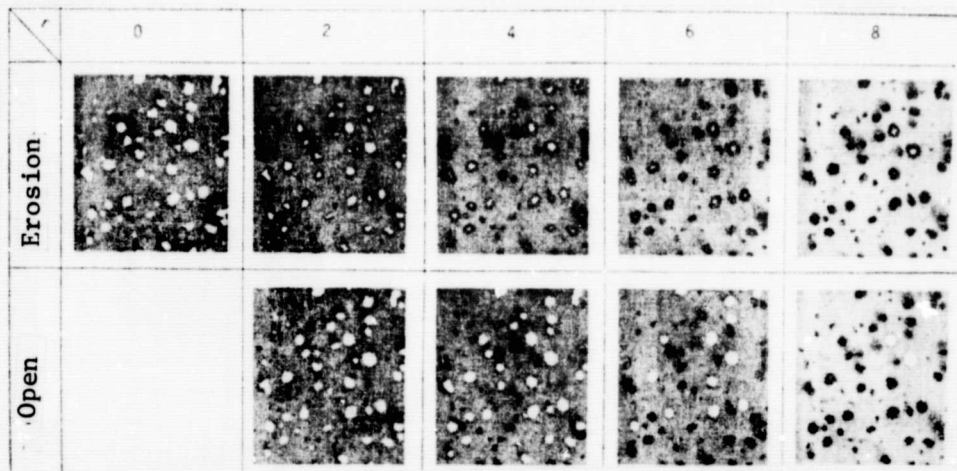
$\Omega_H(X; r=0)$  is the area percentage ( $A_A$ ) of the original image  $X$ .

The above-mentioned equation gives the total particle distribution of particles separated from the open phase. However, the differential particle distribution corresponding to each particle diameter can be obtained from the following equation and becomes as shown in Figure 16 when the distribution for Figure 14 is determined.

$$g_{H(X; r)} = G_{H(r+\delta_r)} - G_{H(r)} \quad (10)$$

Information can be obtained on shape, distribution, and size by tracing the structural elements during image conversion. However, it is also possible to combine different types of image conversion. For instance, new information on distribution can be obtained by erosion by pairs of points after the system has been closed. Moreover, it is also possible to estimate the values for 3-dimensional systems by stereoscopy. Analytical methods that employ image conversion will probably be very useful in the future for application of software to quantitative determination of structures.

(a) Image conversion by open phase



(b) Image conversion by closed phase

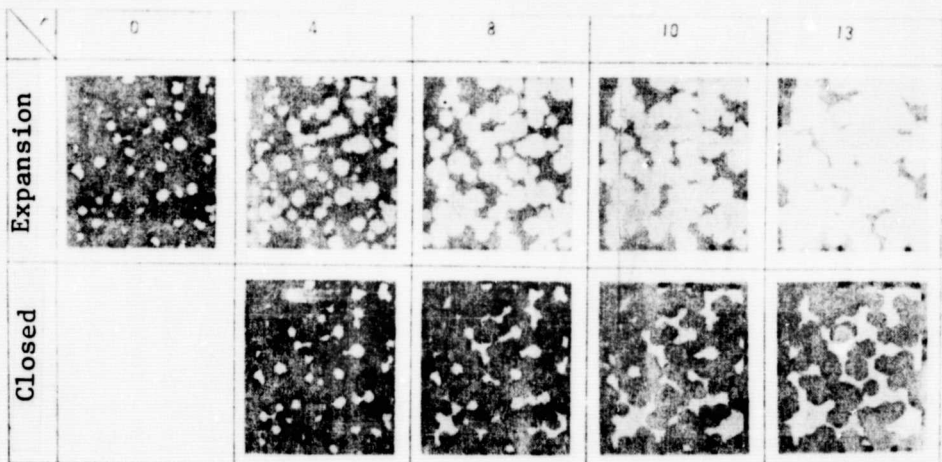


Figure 14. Open (a) and closed (b) phase of spherical graphite by hexagonal structural elements ( $r$  is the size of the structural element and the minimum length is  $2.81 \mu\text{m}$ ).

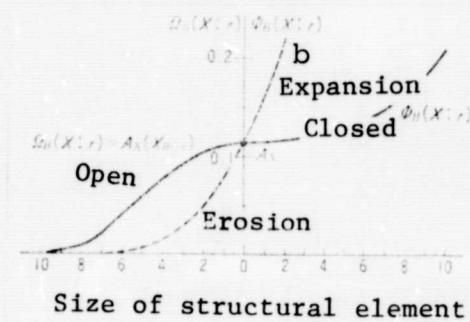


Figure 15. Changes in area percentage by image conversion in Figure 14.

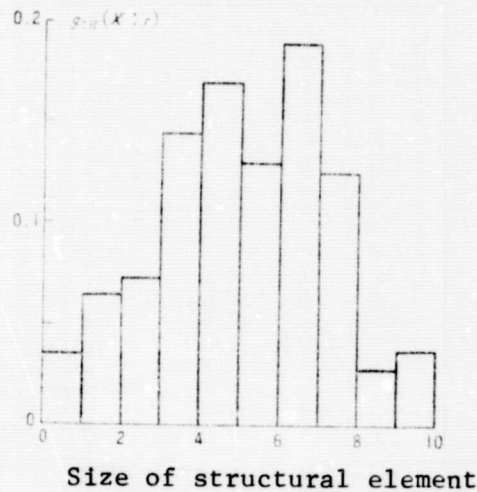


Figure 16. Differential particle distribution from Figure 14(a).



#### 4. Quantitative Determination of Direction

There are many times when the direction of the particles are crystal particles determines the many properties of the material. The fact that special treatment is necessary in stereoscopic treatment of particles that are oriented was previously mentioned. We will show one example of analysis by rose of direction.

Figure 17 is the rose of direction of each crystal particle of the structure in the figure [28]. The rose of direction is represented by  $f=N(a)$  as a function of the degrees taking the number of intercepts in a horizontal direction while rotating the given image by, for instance,  $1^\circ$ . In Figure 17 the direction of each crystal particle is clearly shown. This analytical method is based on measurements of the number of intercepts. Information on the distribution and direction is obtained by simultaneously measuring the rose of direction and covariance for each form of graphite without affecting the shape of the particles.

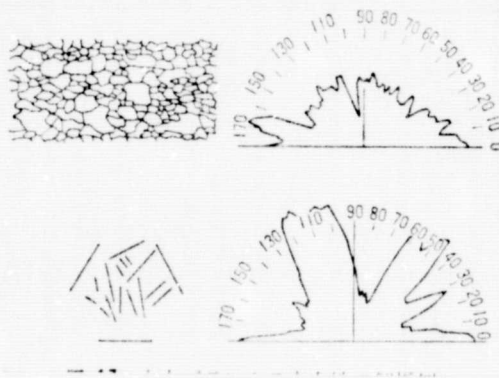


Figure 17. Example of analysis of direction by rose of direction.

#### 5. Conclusion

Case studies were made on analysis of graphite structures in cast iron. The current state with regard to the form, size, distribution and direction of the structure was described. We hope

to use this method to trace the structure of ceramics.

Moreover, quantitative determination of structure is a field of microscopic analysis and various studies have been carried out on the field. Moreover, many different types of software are being developed. In addition, rapid progress is being made in the development of image analyzers, which will promote the use of quantitative microscopy and the development of new materials. Japan also anticipates the establishment of a stereoscopy association.

#### REFERENCES

- [1] H. Elias, "Stereology". Proceeding of the 2nd International Congress for Stereology. H. Elias (ed.) Springer-Verlag, Berlin (1967).
- [2] J. Serra. "Introduction à La Morphologie Mathématique" (Introduction to Mathematical Morphology), Cahiers du Centre de Morphologie Mathématique, Paris (1969).
- [3] Takaku. "Measurements of Form and Analysis and its Application", Research Symposium, Physics Research Institute, 1980, p. 47.
- [4] ISO 945-1975.
- [5] Kimura et al., Waseda University Metals Research Lab Report, Vol. 34, p. 35 (1979).
- [6] Watanabe: "CV Graphite Cast Metal Symposium Text" (Japan Association on Castings, Tokyo) (1982), p. 17.
- [7] Nakada: Konzoku, 48(3) 34(1978).
- [8] Kondo: Imono, 52, 675, (1980).
- [9] V. Kondo, K. Yasue and T. Nishio. Trans Japan Foundrymen's Society, Vol. 1, 62, (1982).
- [10] Kondo: "Measurement and Analysis of Forms and Its Application", Physics Research Symposium, Physics Research Institute (1980), p. 15.
- [11] Kondo: "Measurement and Analysis of Forms and Its Application", (No. 2). Physics Research Symposium, Physics Research Institute (1981) p. 20.

- [12] Kondo: Kinzoku, 49(8), 13 (1979).
- [13] Kondo: Imono, 53, 552(1981).
- [14] Kondo, Kinzoku, '80/3 temporary publication No. 47 (1980).
- [15] Matsuyama, Kinzoku, '81/10 temporary publication No. 47 (1981).
- [16] T.C. Capeletti and J.R. Hornaday, AFS Trans. Vol. 82, 59, (1974).
- [17] Nakada: Imono, 45, 837 (1973).
- [18] Mida: Imono, Vol. 45, p. 563, (1973).
- [19] Nakano: Notes from the 89th Meeting of the Japan Mineral Association, 1976, p. 2.
- [20] Nakada: Notes from the 96th Meeting of the Japan Mineral Assoc., (1979), p. 117.
- [21] Special Subcommittee on Castings of the Japan Association of Minerals, Imono, 40, 296, (1968).
- [22] Osawa: Analysis and Measurement of Forms and Its Application, Physics Research Symposium, Physics Research Institute (1980), p. 39.
- [23] E.E. Underwood, "Quantitative Stereology" (Addison-Wesely) (1970).
- [24] R.T. DeHoff and F.N. Rhines (ed.) "Quantitative Microscopy", McGraw-Hill Book Company, New York (1968).
- [25] Tokushima et al.: "Quantitative Morphology", Uchida Publishers, (1972).
- [26] E. R. Weibel, H. Elias (ed), "Quantitative Methods in Morphology", Springer-Verlag. Berlin (1967).
- [27] Imoto: "Quantitative Morphology", Iwanami Shoten, (1977).
- [28] Ph. Maire and P. Gilles, "Second European Symposium on Quantitative Analysis of Microstructures in Materials Science, Biology and Medicine", Universite de Caen, France (1977) p. 235.

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